REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1. AGENCY USE ONLY (Leave blank)

2. REPORT DATE
January 1990

3. REPORT TYPE AND DATES COVERED
Final Report

4. TITLE AND SUBTITLE
High Temperature Microhardness Tester

5. FUNDING NUMBERS
AFOSR-89-0106 (DURIP)

6. AUTHOR(S)
H.M. Chan

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
Lehigh University
Dept. of Materials Science & Engineering
Whitaker Lab #5
Bethlehem, PA 18015

8. PERFORMING ORGANIZATION REPORT NUMBER
AFOSR.TR. 90-0315

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)
Dr. Liselotte J. Schioler
Air Force Office of Scientific Research
Bolling Air Force Base
Washington, DC 20332

10. SPONSORING/MONITORING AGENCY REPORT NUMBER
61102F
2917/A3

11. SUPPLEMENTARY NOTES

12a. DISTRIBUTION/AVAILABILITY STATEMENT
Unlimited

12b. DISTRIBUTION CODE

13. ABSTRACT (Maximum 200 words)

Funds were obtained through the Defense University Research Instrumentation Program (DURIP) for the purchase of a hardness machine capable of indenting at elevated temperatures. The instrument selected was the Nikon High Temperature Microhardness Tester, Model QM. This apparatus has an operating range from room temperature to 1600°C, with loads varying from 0.5 to 10N. The instrument is now installed and fully operational. It represents a very valuable and convenient method of evaluating the indentation properties of ceramics at elevated temperatures. Although presently at a preliminary stage, the indentation creep studies look particularly promising as a means of studying the high temperature mechanical behavior.

14. SUBJECT TERMS
DURIP

15. NUMBER OF PAGES
12

16. PRICE CODE

17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
Unclassified	Unclassified	Unclassified	Unlimited

Accession For	
NTIS GRA&I	X
DTIC TAB	☐
Unannounced	☐
Justification	
By	
Distribution/	
Availability Codes	
Dist	Avail and/or Special
A-1	

High Temperature Microhardness Tester
Grant # AFOSR-89-0106 (DURIP)

Final Report

Helen M. Chan
Dept. Materials Science & Engineering
Lehigh University
Bethlehem, PA

Introduction

Funds were obtained through the Defense University Research Instrumentation Program (DURIP) for the purchase of a hardness machine capable of indenting at elevated temperatures. The instrument selected was the Nikon High Temperature Microhardness Tester, Model QM. This apparatus has an operating range from room temperature to 1600 ^{o}C, with loads varying from 0.5 to 10 N. An automatic timer enables variation of the load dwell time from 0.1 to 1000s. However if longer indentation times are required, e.g. for indentation creep studies, the time can be controlled manually. The Nikon QM was chosen in preference to other commercially available machines because it can attain significantly higher test temperatures. This was of particular importance, since the rationale for obtaining this apparatus was the need for evaluating the high temperature mechanical properties of ceramic specimens. Furthermore, the Nikon QM was the only instrument in which the indenter and specimen are separately heated, thus ensuring greater temperature control during the indentation process.

90 03 12

Budget

The amount budgeted for the instrument in the original proposal was \$109,032.00 (see Appendix 1). By negotiating the purchase of a demonstration model, Lehigh was able to persuade Nikon to include a temperature control unit (costing \$6,879.00) for the original price. In addition, Nikon agreed to give Lehigh a 5% educational discount (\$5,479.00). Of the remaining cost, \$88,478.00 was provided by the AFOSR (through DURIP), and the balance was provided as cost sharing by Lehigh University.

Installation

The Nikon QM was installed in February 1989, and is located in the Dept. of Materials Science & Engineering, in a laboratory dedicated to that apparatus. A computer controlled log-on system has been installed. This provides detailed records on instrument use time, names of operators etc., and also prevents unauthorized use by untrained persons.

Research

Examples of the types of studies in which the Nikon QM has been utilized are discussed briefly in the following. In terms of the AFOSR project on "Strength and Toughness of Tailored Ceramic Microstructures", the hot hardness tester has been used to evaluate i) the variation of hardness with temperature for a range of $xAl_2O_3.(1-x)c\text{-}ZrO_2$ composites, and ii) indentation creep in Al_2O_3.50 vol% ZrO_2 (AZ50). The results of these tests are depicted in Figures 1 and 2 respectively. Figure 1 shows that as the c-ZrO_2 content increases, the hardness of the composite (at any given temperature) decreases. It is interesting, however, that all of the hardness vs. temperature curves show a discontinuity in the slope at ~ 500 ^{o}C, indicating a change in the deformation mechanism. Figure 2 shows (somewhat intriguingly) that although single phase alumina is harder than single phase cubic zirconia, the duplex AZ50 is harder at 1200 ^{o}C for all indentation times. Work is currently underway to determine the underlying physical mechanisms for this behavior.

Data taken using the Nikon QM in related studies are shown in Figures 3 and 4. Figure 3 shows indentation creep results for polycrystalline cubic zirconia. These results, together with indentation creep data for Al_2O_3 will be compared with that of AZ50 to gain an estimate of the relative creep resistance of the duplex material. Figure 4 shows a comparison of the high temperature hardness behavior of anorthite ($CaO.Al_2O_3.2SiO_2$) and alumina. It can be seen that although at room temperature the hardness of anorthite is significantly lower than that of alumina, at around 1000 ^{o}C, the hardness values are very much comparable. This information proved to be very valuable in interpreting the mechanical properties of a two-phase alumina in which the intergranular glassy phase had been crystallized to form anorthite. Finally, Figure 5 is an example of work taken from a project related to electronic packaging materials, and shows the temperature dependence of hardness of AlN.

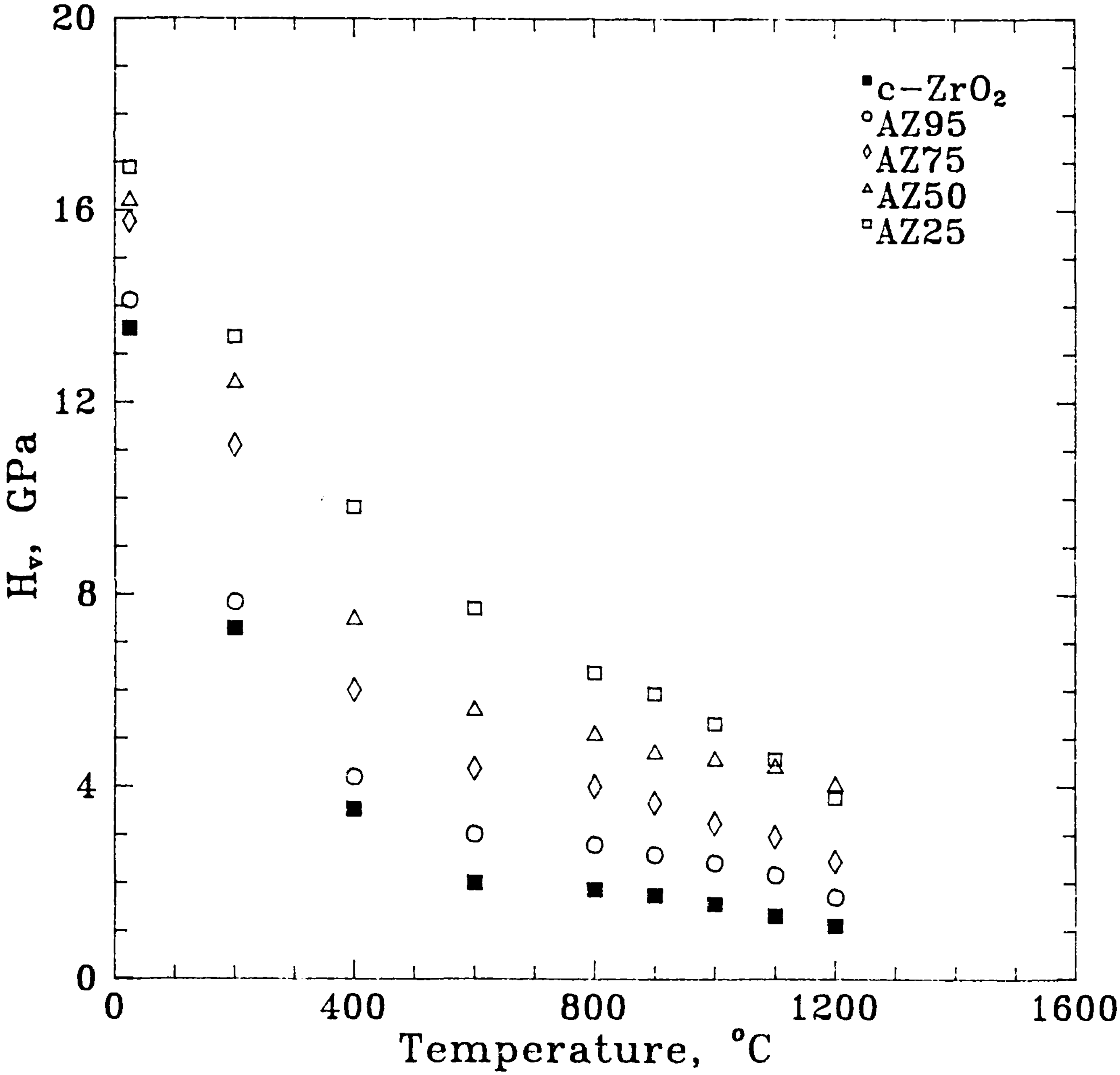

Figure 1

Graphs of Vickers hardness vs. temperature for a series of x Al_2O_3 . (1-x) c-ZrO_2 composites (x = 0, .05, .25, .5 and .75).

Courtesy J.D. French

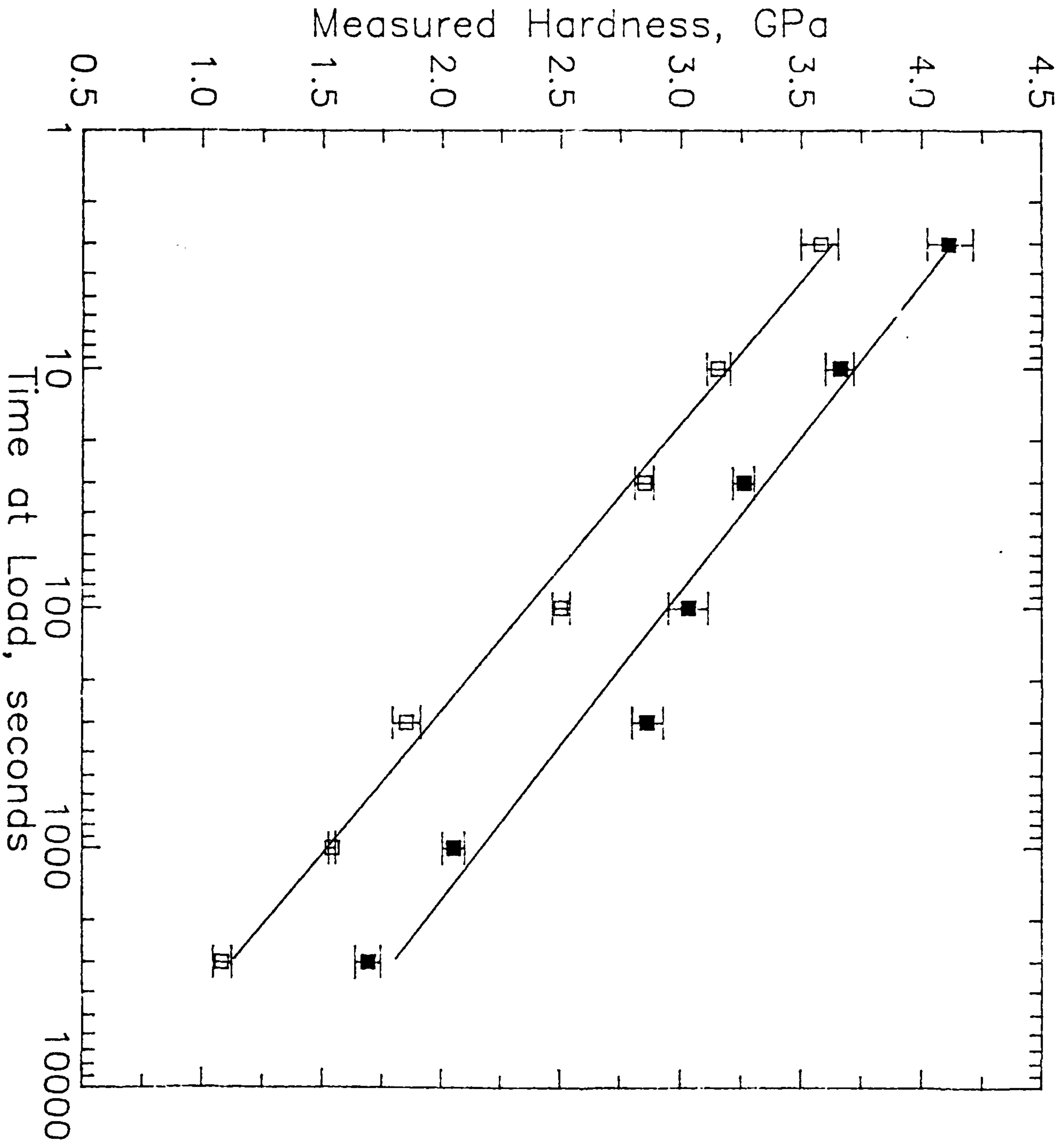

Figure 2

Indentation creep curves at 1200°C for AZ50 (filled symbols) and Al_2O_3 (open symbols)

Courtesy J.D. French

Creep Studies for Polycrystalline Zirconia @ 26 C, 500 C and 1000 C

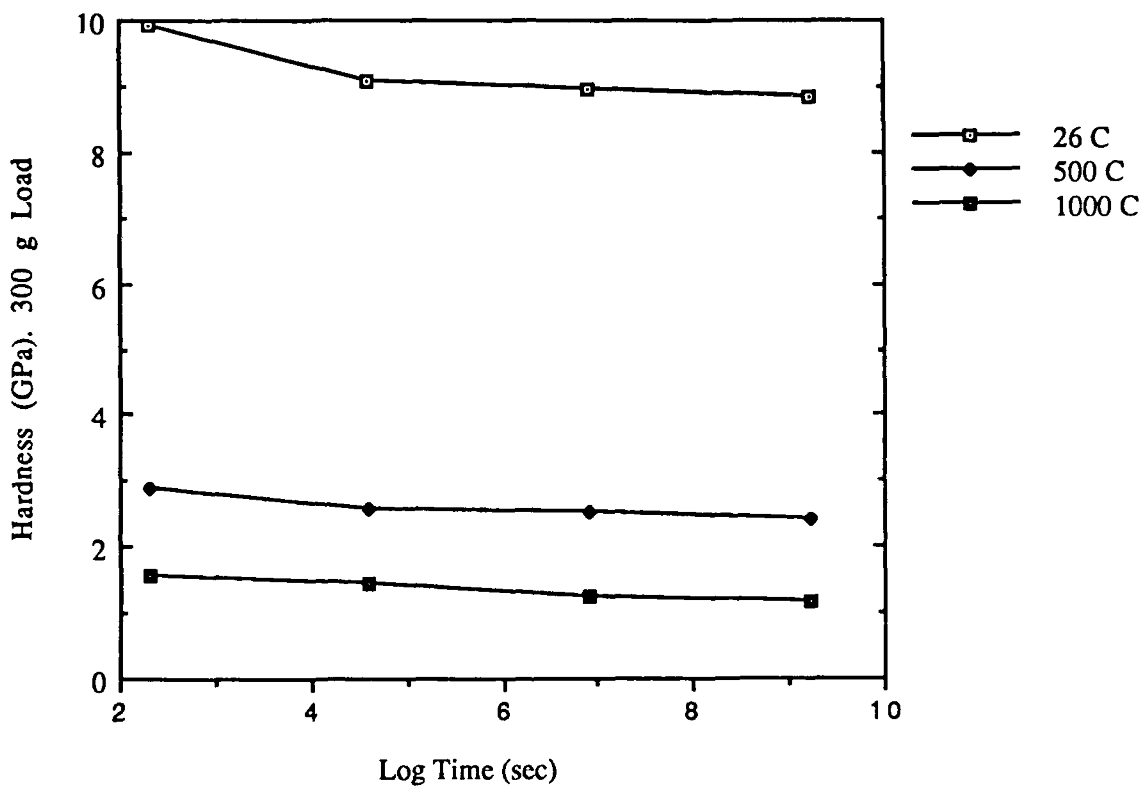

Figure 3

Indentation creep curves for c-ZrO_2 obtained at 26°C, 500°C and 1000°C.

Courtesy J. Stanescu

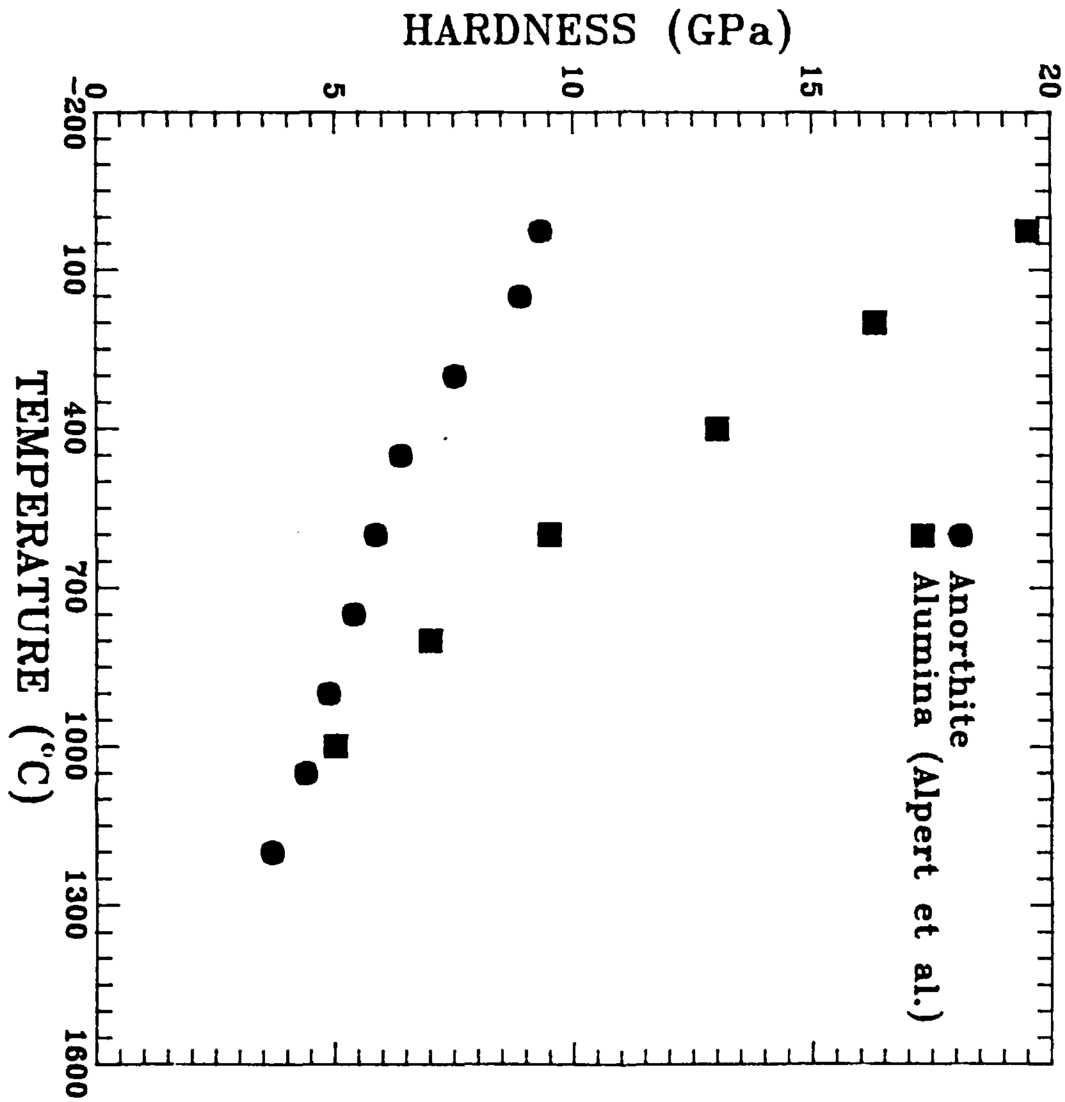

Figure 4

Graphs of Vickers hardness vs. temperature for anorthite and alumina.

Courtesy N.P. Padture

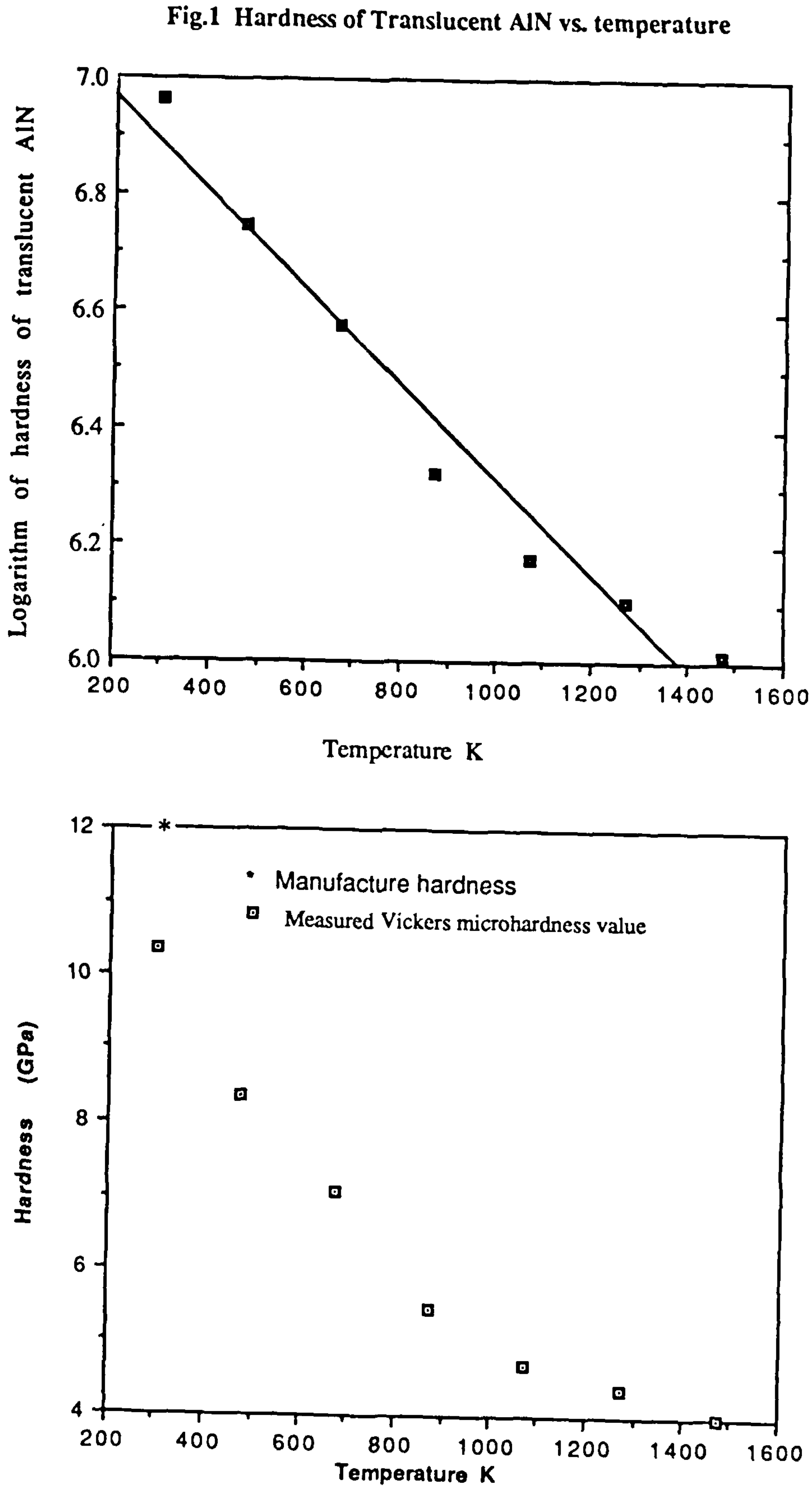

Figure 5
Graphs of Vickers hardness vs. temperature for AlN.
Courtesy T. Liu and M.R. Notis

The list of projects at Lehigh which currently benefit from the apparatus is summarized in Table 1. In addition to usuage by Lehigh faculty and students, there have been several external users of the facility, and these are listed in Table II.

Summary

The Nikon QM high temperature microhardness tester is now installed and fully operational. It represents a very valuable and convenient method of evaluating the indentation properties of ceramics at elevated temperatures. Although presently at a preliminary stage, the indentation creep studies look particularly promising as a means of studying the high temperature mechanical behavior. As research progresses, it is intended to combine this data with detailed microstructural examination of the indentation sites. It is envisaged that this will yield much needed information on the influence of temperature on the deformation mechanisms in ceramics.

TABLE I

Current DoD and DoD Related Research Benefitting
From High Temperature Microhardness Capability

Contract	Title	Principal Investigators	Current Year Funding	Continuing Funding
AFOSR #870396	Strength and Toughness of Tailored Ceramic Micro-Structures	M P. Harmer H.M. Chan G.A. Miller	$190,585	Through 10/90
DMR-8920844	Influence of Temperature on Indentation-Induced Flow Nucleation Processes in Ceramic Materials	H.M. Chan	$100,000	Through 6/91
Coors	R-Curve Behavior of Glass Ceramics	H.M. Chan B.R. Lawn M.J. Readey	$40,000	Through 6/91
DE-FG02-86ER45256	Grain Boundary Diffusion in Oriented Ni_3Al Bicrystals Containing Boron	Y.T. Chou	$89,500	Through 9/90
DE-FG02-84ER45150	Analytical Electron Micro-scopy Studies of Interfaces and Phase Transformations in Zirconia Ceramic Systems	M.R. Notis	$118,000	Through 8/90
Semiconductor Research Corp.	Electronic Device Packaging Technology: Sub Program in New Ceramic Substrate Materials	M.R. Notis (R.J. Jaccodine)	$40,000	Through 12/90
DMR-8905459	Quantitative Microanalysis of Li and Be Intermetallics		$113,000	Through 6/92

TABLE II

External Users o. High Temperature Micrchardness Facility

1. Ms. S.W. Wang and Dr. A. Majidi
   Dept. Mechanical Engineering
   University of Delaware
   Project Title: "Processing and Characterization of Continuous Fiber Reinforced Glass and Glass-Ceramic Matrix Composites"
   Sponsored by AFOSR

2. Dr. C.D. Graham
   Dept. Materials Science and Engineering
   University of Pennsylvania
   Project Title: "Development of Texture by Warm Deformation of Fe-Nd-B Permanent Magnets"
   Sponsored by NSF

3. SCM Metal Products, Inc.
   11000 Cedar Avenue Suite 100
   Cleveland, OH 44106

4. Dr. S. Bose
   United Technologies
   Pratt and Whitney
   E. Hartford, CT 06108

APPENDIX I

BUDGET

Nikon High Temperature Microhardness Tester Model QM	$109,032.00

Basic Unit consisting of

- Indenter loading and vacuum control unit
- Heating control unit
- Power control unit
- Console desk equipped with micrometer
- Lamp housing
- Rotary oil pump
- Supplied with:
  - 2 high temperature furnaces
  - 2 furnaces for indenter
  - 2 specimen holder (cylindrical)
  - 2 specimen holder (block format)
  - 2 diamond micro-vickers indenter
  - 2 sapphire micro-vickers indenter
  - 2 halogen lamps (12V/100W)

Accessories	
Diamond knoop indenter	539.00
TOTAL	$109,571.00
Educational Discount (5%)	- 5,479.00
	$104,092.00
Less cost sharing by Lehigh University (15%)	-15,614.00
Amount Requested from DoD	$ 88,478.00

Contact Person: Mr. Matthew Smith
Nikon Inc.
(516) 222-0200